

### **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

### **Order Summary Report**

Order Number:	J13030193				
Project Name:	WWTS - Biweekly (2)				
Customer Name(s):	Bill K., Ron L., Don S., Ray L.				
Customer Address:	253 Plant Allen Road				
	Belmont, NC 28012				
Lab Contact:	Jason C Perkins	Phone:	980-875-5348		
Report Authorized By:		Dat	e:	3/28/2013	
(Signature)	Jason C Perkins				

#### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any guestions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013005716	ALLEN	08-Mar-13 6:37 AM	C. MCHUGH	FGD Purge Eff
2013005717	ALLEN	08-Mar-13 6:40 AM	C. MCHUGH	EQ Tank Eff
2013005718	ALLEN	08-Mar-13 6:44 AM	C. MCHUGH	BioReactor 1 Inf
2013005719	ALLEN	08-Mar-13 6:55 AM	C. MCHUGH	BioReactor 2 Inf
2013005720	ALLEN	08-Mar-13 6:50 AM	C. MCHUGH	BioReactor 2 Eff
2013005721	ALLEN	08-Mar-13 8:20 AM	C. MCHUGH	Filter Blk
2013005722	ALLEN	18-Dec-13 9:00 AM	C. KNOX	TRIP BLANK
2013005723	ALLEN	08-Mar-13 7:20 AM	C. MCHUGH	HG BioReactor 1 Inf
2013005724	ALLEN	08-Mar-13 7:20 AM	C. MCHUGH	Hg Blk BioReactor 1 Inf
2013005725	ALLEN	08-Mar-13 7:27 AM	C. MCHUGH	Hg BioReactor 2 Inf
2013005726	ALLEN	08-Mar-13 7:27 AM	C. MCHUGH	Hg Blk BioReactor 2 Inf
2013005727	ALLEN	08-Mar-13 7:24 AM	C. MCHUGH	Hg BioReactor 2 Eff
2013005728	ALLEN	08-Mar-13 7:24 AM	C. MCHUGH	Hg Blk BioReactor 2 Eff

### **Technical Validation Review**

### **Checklist:**

COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures).

All Results are less than the laboratory reporting limits. □ Yes ✓ No

All laboratory QA/QC requirements are acceptable. ✓ Yes □ No

### **Report Sections Included:**

Reviewed By:

**DBA Account** 

✓ Job Summary Report	✓ Sub-contracted Laboratory Results
✓ Sample Identification	$\hfill\Box$ Customer Specific Data Sheets, Reports, & Documentation
✓ Technical Validation of Data Package	☐ Customer Database Entries
✓ Analytical Laboratory Certificate of Analysis	✓ Chain of Custody
☐ Analytical Laboratory QC Report	✓ Electronic Data Deliverable (EDD) Sent Separately

Date:

3/28/2013

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### Order # J13030193

Site: FGD Purge Eff Sample #: 2013005716

Collection Date: 08-Mar-13	6:37 AM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	720	mg/L		10	100	EPA 300.0	03/25/2013 08:12	BGN9034
MERCURY (COLD VAPOR) IN W	<u>VATER</u>							
Mercury (Hg)	39.1	ug/L		2.5	50	EPA 245.1	03/21/2013 13:14	AGIBBS
TOTAL RECOVERABLE METAL	S BY ICP							
Boron (B)	84.2	mg/L		0.5	10	EPA 200.7	03/19/2013 13:19	MHH7131
DISSOLVED METALS BY ICP-M	<u>IS</u>							
Selenium (Se)	3760	ug/L		10	10	EPA 200.8	03/22/2013 12:18	KRICHAR
TOTAL RECOVERABLE METAL	S BY ICP-MS							
Arsenic (As)	192	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
Chromium (Cr)	151	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
Copper (Cu)	191	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
Nickel (Ni)	218	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
Selenium (Se)	10600	ug/L		50	50	EPA 200.8	03/19/2013 11:31	KRICHAR
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
Zinc (Zn)	262	ug/L		10	10	EPA 200.8	03/19/2013 11:31	KRICHAR
SELENIUM SPECIATION - (Anal	lysis Performed b	y Applied	Speciation a	nd Consu	ulting, LL	<u>-C)</u>		
Vendor Parameter	Complete					Vendor Metho	od	V_AS&C
TOTAL DISSOLVED SOLIDS								
TDS	8200	mg/L		200	1	SM2540C	03/19/2013 15:35	SWILLI3
Site: EQ Tank Eff						Sample #:	2013005717	
Collection Date: 08-Mar-13	6:40 AM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY (COLD VAPOR) IN W	<u>VATER</u>							
Mercury (Hg)	35.1	ug/L		2.5	50	EPA 245.1	03/21/2013 13:17	AGIBBS

Mercury (Hg) EPA 245.1 03/21/2013 13:17 AGIBBS TOTAL RECOVERABLE METALS BY ICP Boron (B) 0.5 10 EPA 200.7 03/19/2013 13:23 MHH7131 90.5 mg/L **DISSOLVED METALS BY ICP-MS** Selenium (Se) 4300 ug/L 10 10 EPA 200.8 03/22/2013 12:21 KRICHAR

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### Order # J13030193

Site: EQ Tank Eff

Collection Date: 08-Mar-13 6:40 AM

Sample #: 2013005717

Matrix: OTHER

Analyte	Result	Units (	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METAL								
Arsenic (As)	166	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR
Chromium (Cr)	126	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR
Copper (Cu)	161	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR
Nickel (Ni)	192	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR
Selenium (Se)	9930	ug/L		50	50	EPA 200.8	03/19/2013 11:35	KRICHAR
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR
Zinc (Zn)	227	ug/L		10	10	EPA 200.8	03/19/2013 11:35	KRICHAR

Site: BioReactor 1 Inf Sample #: 2013005718

Collection Date: 08-Mar-13 6:44 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
TOTAL RECOVERABLE METALS BY ICP										
Boron (B)	120	mg/L		0.5	10	EPA 200.7	03/19/2013 13:27	MHH7131		
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	3750	ug/L		10	10	EPA 200.8	03/22/2013 12:25	KRICHAR		
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Nickel (Ni)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Selenium (Se)	3980	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	03/19/2013 11:38	KRICHAR		

### SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)

Vendor Parameter Complete Vendor Method V\_AS&C

Site: BioReactor 2 Inf Sample #: 2013005719

Collection Date: 08-Mar-13 6:55 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	S BY ICP							
Boron (B)	153	mg/L		0.5	10	EPA 200.7	03/19/2013 13:31	MHH7131

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### Order # J13030193

Site: BioReactor 2 Inf

Sample #:

2013005719

Collection Date: 08-Mar-13 6:55 AM

Matrix:

OTHER

Analyte	Result	Units Qualifie	ers RDL	DF	Method	Analysis Date/Time	Analyst	
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Chromium (Cr)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Copper (Cu)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Nickel (Ni)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Selenium (Se)	120	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Silver (Ag)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	
Zinc (Zn)	< 10	ug/L	10	10	EPA 200.8	03/19/2013 11:42	KRICHAR	

Site: BioReactor 2 Eff

Sample #:

2013005720

Collection Date: 08-Mar-13 6:50 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst	
INORGANIC IONS BY IC									
Bromide	210	mg/L		5	50	EPA 300.0	03/22/2013 15:29	BGN9034	
MERCURY (COLD VAPOR) IN WATER									
Mercury (Hg)	< 1	ug/L		1	20	EPA 245.1	03/21/2013 13:19	AGIBBS	
TOTAL RECOVERABLE METALS BY ICP									
Boron (B)	215	mg/L		0.5	10	EPA 200.7	03/19/2013 13:35	MHH7131	
TOTAL RECOVERABLE METALS BY	Y ICP-MS								
Arsenic (As)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Chromium (Cr)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Copper (Cu)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Nickel (Ni)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Selenium (Se)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Silver (Ag)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	
Zinc (Zn)	< 5	ug/L		5	5	EPA 200.8	03/19/2013 11:45	KRICHAR	

### SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)

Vendor Parameter Complete Vendor Method V\_AS&C

Site: Filter Blk Sample #: 2013005721

Collection Date: 08-Mar-13 8:20 AM

Matrix: OTHER

Units Qualifiers RDL Analysis Date/Time **Analyte** Result DF Method Analyst **DISSOLVED METALS BY ICP-MS** Selenium (Se) 1.70 EPA 200.8 03/22/2013 11:59 **KRICHAR** ug/L 1

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### Order # J13030193

Site: TRIP BLANK 2013005722 Sample #: OTHER Collection Date: 18-Dec-13 9:00 AM Matrix: Analyte Result Units Qualifiers **RDL** DF Method **Analysis Date/Time** Analyst **TOTAL RECOVERABLE METALS BY ICP** Boron (B) 0.05 EPA 200.7 03/19/2013 12:52 < 0.05 mg/L 1 MHH7131 **TOTAL RECOVERABLE METALS BY ICP-MS** EPA 200.8 Arsenic (As) < 1 ug/L 1 1 03/19/2013 11:07 **KRICHAR** Chromium (Cr) < 1 ug/L 1 EPA 200.8 03/19/2013 11:07 **KRICHAR** Copper (Cu) < 1 ug/L 1 EPA 200.8 03/19/2013 11:07 **KRICHAR** Nickel (Ni) ug/L 1 EPA 200.8 03/19/2013 11:07 **KRICHAR** < 1 Selenium (Se) < 1 ua/L EPA 200.8 03/19/2013 11:07 **KRICHAR** Silver (Ag) < 1 ug/L 1 EPA 200.8 03/19/2013 11:07 **KRICHAR** Zinc (Zn) < 1 ug/L 1 EPA 200.8 03/19/2013 11:07 **KRICHAR** SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC) Vendor Parameter Complete Vendor Method V\_AS&C Site: HG BioReactor 1 Inf Sample #: 2013005723 Collection Date: 08-Mar-13 7:20 AM Matrix: **OTHER** Analyte Result Units Qualifiers **RDL** DF Method **Analysis Date/Time** Analyst MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC) Vendor Method V\_BRAND Vendor Parameter Complete Site: Hg Blk BioReactor 1 Inf Sample #: 2013005724 Collection Date: 08-Mar-13 7:20 AM Matrix: OTHER Analyte Result Units Qualifiers **RDL** DF Method **Analysis Date/Time** Analyst MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC) Vendor Parameter V\_BRAND Complete Vendor Method Site: Hg BioReactor 2 Inf Sample #: 2013005725 Collection Date: 08-Mar-13 7:27 AM Matrix: OTHER Analyte Result Units Qualifiers RDL Method **Analysis Date/Time** Analyst MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC) Vendor Parameter Complete Vendor Method V\_BRAND

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#### Order # J13030193

Site: Hg Blk BioReactor 2 Inf Sample #: 2013005726

Collection Date: 08-Mar-13 7:27 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC)

Vendor Parameter Complete Vendor Method V\_BRAND

Site: Hg BioReactor 2 Eff Sample #: 2013005727

Collection Date: 08-Mar-13 7:24 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC)

Vendor Parameter Complete Vendor Method V\_BRAND

Site: Hg Blk BioReactor 2 Eff Sample #: 2013005728

Collection Date: 08-Mar-13 7:24 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

MERCURY 1631 - (Analysis Performed by Brooks Rand Labs LLC)

Vendor Parameter Complete Vendor Method V\_BRAND



March 27, 2013

Duke Energy ATTN: Jay Perkins Scientific Support-Laboratory 13339 Hagers Ferry Road Huntersville NC 28078 jcperkins@duke-energy.com labcustomer@duke-energy.com

RE: Project DUK-HV1201 Client Project: J13030193

Dear Mr. Perkins,

On March 15, 2013, Brooks Rand Labs (BRL) received three (3) waste water samples and three (3) field blanks samples. The samples were logged-in for total mercury (Hg) analysis. All samples were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the relevant SOP and may have been evaluated using reporting limits that have been adjusted to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific MDLs, MRLs, and other details.

All field blanks yielded detectable results; however, all results were less than the method defined control limit of 0.5 ng/L and were considered insignificant sources of contamination. All data was reported without further qualification, aside from concentration qualifiers, and all associated quality control sample results met the acceptance criteria.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report.

Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com



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### Report Information

#### **Laboratory Accreditation**

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

### **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

#### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	Т	total recoverable fraction

#### **Definition of Data Qualifiers**

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- **J-M** Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- M Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- **X** Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand Labs, those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review; USEPA; January 2010. These supersede all previous qualifiers ever employed by BRL.</u>



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# Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1311019-01	Influent	Sample	03/08/2013	03/15/2013
Hg Blk BioReactor 1 Inf	1311019-02	DIW	Field Blank	03/08/2013	03/15/2013
BioReactor 2 Inf	1311019-03	Influent	Sample	03/08/2013	03/15/2013
Hg Blk BioReactor 2 Inf	1311019-04	DIW	Field Blank	03/08/2013	03/15/2013
BioReactor 2 Eff	1311019-05	Effluent	Sample	03/08/2013	03/15/2013
Hg Blk BioReactor 2 Eff	1311019-06	DIW	Field Blank	03/08/2013	03/15/2013

# **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	03/18/2013	03/20/2013	B130434	1300194



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# Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1311019-01	<b>nf</b> Hg	Influent	T	433		0.77	2.04	ng/L	B130434	1300194
<b>BioReactor 2 E</b> 1311019-05	<b>ff</b> Hg	Effluent	Т	20.8		0.15	0.41	ng/L	B130434	1300194
<b>BioReactor 2 In</b> 1311019-03	h <b>f</b> Hg	Influent	Т	96.0		0.15	0.41	ng/L	B130434	1300194
<b>Hg Blk BioRead</b> 1311019-02	c <b>tor 1 Inf</b> Hg	DIW	T	0.42		0.15	0.40	ng/L	B130434	1300194
<b>Hg Blk BioRead</b> 1311019-06	c <b>tor 2 Eff</b> Hg	DIW	T	0.40	В	0.15	0.41	ng/L	B130434	1300194
<i>Hg Blk BioRead</i> 1311019-04	ctor 2 Inf Hg	DIW	Т	0.37	В	0.15	0.40	ng/L	B130434	1300194



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# Accuracy & Precision Summary

Batch: B130434 Lab Matrix: Water Method: EPA 1631

Sample B130434-SRM1	Analyte Certified Reference Materia	Native al (1310011	Spike , NIST 1641d	Result	Units	REC & Limits	RPD & Limits
	Hg	·	15.68	15.58	ng/L	99% 75-125	
B130434-MS2	<b>Matrix Spike (1311004-01)</b> Hg	221.2	1263	1318	ng/L	87% 71-125	
B130434-MSD2	Matrix Spike Duplicate (13 <sup>4</sup>	1 <b>1004-01)</b> 221.2	1263	1415	ng/L	95% 71-125	7% 24

## Method Blanks & Reporting Limits

Batch: B130434 Matrix: Water Method: EPA 1631 Analyte: Hg

Sample	Result	Units
B130434-BLK1	0.21	ng/L
B130434-BLK2	0.22	ng/L
B130434-BLK3	0.33	ng/L
B130434-BLK4	0.20	ng/L

 Average: 0.24
 Standard Deviation: 0.06
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.41



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## **Instrument Calibration**

Sequence: 1300194 Total Mercury and Mercury Speciation by CVAFS

Method: EPA 1631

Instrument: THG-06 Date: 03/20/2013 Analyte: Hg

<b>Lab ID</b> 1300194-IBL1	True Value	Result 3.23	Units pg of Hg	REC	& Limits
1300194-IBL2		3.95	pg of Hg		
1300194-IBL3		5.38	pg of Hg		
1300194-IBL4		5.99	pg of Hg		
1300194-CAL1	10.00	11.55	pg of Hg	115%	
1300194-CAL2	25.00	25.16	pg of Hg	101%	
1300194-CAL3	100.0	99.72	pg of Hg	100%	
1300194-CAL4	500.0	481.8	pg of Hg	96%	
1300194-CAL5	2500	2434	pg of Hg	97%	
1300194-CAL6	10000	9322	pg of Hg	93%	
1300194-ICV1	1568	1558	pg of Hg	99%	85-115
1300194-CCB1		8.06	pg of Hg		
1300194-CCV1	500.0	498.4	pg of Hg	100%	77-123
1300194-CCB2		7.41	pg of Hg		
1300194-CCB3		7.66	pg of Hg		
1300194-CCB4		7.23	pg of Hg		
1300194-CCV2	500.0	496.2	pg of Hg	99%	77-123
1300194-CCB5		6.78	pg of Hg		
1300194-CCV3	500.0	496.0	pg of Hg	99%	77-123
1300194-CCB6		6.96	pg of Hg		
1300194-CCV4	500.0	494.8	pg of Hg	99%	77-123
1300194-CCB7		7.24	pg of Hg		
1300194-CCV5	500.0	498.3	pg of Hg	100%	77-123
1300194-CCB8		6.79	pg of Hg		
1300194-CCV6	500.0	484.5	pg of Hg	97%	77-123
1300194-CCB9		6.49	pg of Hg		
1300194-CCV7	500.0	476.7	pg of Hg	95%	77-123
1300194-CCBA		5.77	pg of Hg		
1300194-CCV8	500.0	481.0	pg of Hg	96%	77-123
1300194-CCBB		6.21	pg of Hg		
1300194-CCV9	500.0	479.0	pg of Hg	96%	77-123
1300194-CCBC		6.14	pg of Hg		
1300194-CCVA	500.0	481.4	pg of Hg	96%	77-123
1300194-CCBD		5.75	pg of Hg		
1300194-CCVB	500.0	479.2	pg of Hg	96%	77-123
1300194-CCBE		6.15	pg of Hg		
1300194-CCVC	500.0	472.7	pg of Hg	95%	77-123
1300194-CCBF		6.26	pg of Hg		
1300194-CCVD	500.0	475.0	pg of Hg	95%	77-123
1300194-CCBG		6.13	pg of Hg		



Page 15 of 28 Client PM: Jay Perkins Client PO: 141391

## **Instrument Calibration**

Sequence: 1300194 Total Mercury and Mercury Speciation by CVAFS

Method: EPA 1631

**Instrument:** THG-06 **Date:** 03/20/2013

Analyte: Hg

Lab ID	True Value	Result	Units	REC	C & Limits
1300194-CCVE	500.0	474.8	pg of Hg	95%	77-123
1300194-CCBH		5.59	pg of Hg		
1300194-CCVF	500.0	482.9	pg of Hg	97%	77-123
1300194-CCBI		5.63	pg of Hg		
1300194-CCVG	500.0	479.3	pg of Hg	96%	77-123
1300194-CCBJ		5.51	pg of Hg		
1300194-CCVH	500.0	494.4	pg of Hg	99%	77-123
1300194-CCBK		6.08	pg of Hg		
1300194-CCVI	500.0	498.7	pg of Hg	100%	77-123
1300194-CCBL		7.30	pg of Hg		
1300194-CCVJ	500.0	496.1	pg of Hg	99%	77-123
1300194-CCBM		5.76	pg of Hg		
1300194-CCVK	500.0	494.3	pg of Hg	99%	77-123
1300194-CCBN		6.17	pg of Hg		
1300194-CCVL	500.0	486.1	pg of Hg	97%	77-123
1300194-CCBO		6.11	pg of Hg		
1300194-CCVM	500.0	493.1	pg of Hg	99%	77-123
1300194-CCBP		5.92	pg of Hg		



Page 16 of 28 Client PM: Jay Perkins Client PO: 141391

# **Sample Containers**

	ID: 1311019-01 ple: BioReactor 1 Inf		•	rt Matrix: Influent ble Type: Sample			cted: 03/08/2013 ived: 03/15/2013
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71691270 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler
	ID: 1311019-02 ple: Hg Blk BioReactor 1 Inf		•	rt Matrix: DIW ble Type: Field Blank			cted: 03/08/2013 ived: 03/15/2013
<b>Des</b> A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71691270 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler
	ID: 1311019-03 ple: BioReactor 2 Inf		•	rt Matrix: Influent			cted: 03/08/2013 ived: 03/15/2013
<b>Des</b> A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71691270 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler
	ID: 1311019-04 ple: Hg Blk BioReactor 2 Inf		•	rt Matrix: DIW Die Type: Field Blank			cted: 03/08/2013 ived: 03/15/2013
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71691270 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler
	ID: 1311019-05 ple: BioReactor 2 Eff		-	rt Matrix: Effluent ble Type: Sample			cted: 03/08/2013 ived: 03/15/2013
<b>Des</b> A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71691270 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler
	ID: 1311019-06 ple: Hg Blk BioReactor 2 Eff		•	rt Matrix: DIW ble Type: Field Blank			cted: 03/08/2013 ived: 03/15/2013
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71691270 10	Preservation none	<b>P-Lot</b> n/a	рН	Ship. Cont. Cooler



Page 17 of 28 Client PM: Jay Perkins Client PO: 141391

# **Shipping Containers**

Cooler

**Received:** March 15, 2013 8:50 **Tracking No:** 557566432441 via FedEx

Coolant Type: Ice Temperature: -0.5 °C Description: Cooler
Damaged in transit? No
Returned to client? No

Custody seals present? No Custody seals intact? No COC present? Yes

Customer to complete all appropriate non-shaded areas.  LAB USE ONLY  Se Speciation Bottlar  10
Special Color   Special Colo
### See Speciation Bottlet ### ### #### #### ################
23   BioReactor 1 Inf 3-9-13 0720 CSM BAC   1   24   Hg Bik BioReactor 1 Inf 3-9-13 0720 CSM BAC   1   1   25   BioReactor 2 Inf 3-9-13 0727 CSM BAC   1   1   1   1   1   1   1   1   1
1   Hg Blk BioReactor 2 Inf   35/13 0727 C5/h (3/h)   1   27
Hg Bik BioReactor 2 Eff 5-5-15-0724 CSm Byte 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

March 21, 2013

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Allen - FGD WWTS (2010, Bi-Monthly Sampling) (LIMS #J13030193)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation on March 14, 2013. The samples were received in a sealed cooler at -0.5°C on March 15, 2013. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Allen - FGD WWTS (2010, Bi-Monthly Sampling) (LIMS #J13030193)

March 21, 2013

### 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on March 14, 2013. The samples were received on March 15, 2013 in a sealed container at -0.5°C.

The samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45μm) and injected directly into an autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of

each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on March 16, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with the samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

# Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (2010, Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J13030193

Date: March 21, 2013 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	4170	1560	217	ND (< 3.0)	ND (< 3.0)	117 (3)
BioReactor 1 Inf	1440	1560	120	3.83	ND (< 0.75)	85.6 (3)
BioReactor 2 Eff	1.26	ND (<1.1)	ND (< 0.60)	ND (< 0.75)	ND (< 0.75)	0.0 (0)
Metals Trip Blk	ND (< 0.023)	ND (< 0.043)	ND (< 0.024)	ND (< 0.030)	ND (< 0.030)	0.0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

# Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (2010, Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J13030193

Date: March 21, 2013 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 250x	eMDL 1000x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	0.023	0.57	2.3
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.004	0.043	1.1	4.3
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.002	0.024	0.60	2.4
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.030	0.75	3.0
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.030	0.75	3.0

eMDL = Estimated Method Detection Limit

### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	9.123	95.3
Se(VI)	LCS	9.48	8.707	91.8
SeCN	LCS	8.92	8.484	95.1
MeSe(IV)	LCS	6.47	6.152	95.1
SeMe	LCS	9.32	8.709	93.4

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (2010, Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J13030193

Date: March 21, 2013 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	Batch QC	ND (< 2.3)	ND (< 2.3)	NC	NC
Se(VI)	Batch QC	458.6	434.9	446.7	5.3
SeCN	Batch QC	ND (< 2.4)	ND (< 2.4)	NC	NC
MeSe(IV)	Batch QC	ND (< 3.0)	ND (< 3.0)	NC	NC
SeMe	Batch QC	ND (< 3.0)	ND (< 3.0)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

### Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	Batch QC	5560	5287	95.1	5560	5194	93.4	1.8
Se(VI)	Batch QC	5045	5127	92.8	5045	5116	92.6	0.2
SeCN	Batch QC	4575	4252	92.9	4575	4158	90.9	2.2

	Duke Energy Analytical Laboratory	ytical Laboratory Use Only			Ana	ytical La	Analytical Laboratory Use Only	Use Or	ķ		•	
Duke	Mail Code MGO3A	Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd	- Alms	3030	13030193 MATRIX OTHER	MATRIX OT		Samples Originating	iles nating	SC	Page 1 of 2	Page 1 of 2
Agual	Huntersville, (704) 87 (704) 87 (704)	Huntersville, N. C. 28078 (704) 875-5245 Fax: (704) 875-4349	Logged By	-9	Date & Time	5	10	m	SAMPLE PROGRAM	GRAM Ground NPDES	ORIGINAL to LAB,	L to LAB, CLIENT
1)Project Name	Allen - FGD WWTS (2010, Bl-Monthly Sampling)	2)Phone No:	Vendor	AS&C		og S	Cooler Temp (C		RCRA Waste	Drinking Water UST Waste	9	
2) Cilent: Don Scri	Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy	4)Fax No:				2=H,SO,	Preserv::1=HCL =H,SO <sub>4</sub> 3=HNO <sub>3</sub>		7			370
5)Business Unit:	6)Process:	Mail Code:	MR#				ses			()	pell	
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"Lab ID ID		13 Sample Description or ID	Date	Time	Sign	Signature	pmoට <sup>71</sup> ර්තව <sup>81</sup>	SQT	Br (Di	s 'ac	AS&C	3
18205716	FGD	FGD Purge Eff	3-8-13	0637	CSM	BM	7		1		•	
1110	EQ.	EQ Tank Eff.	3-8-13	0640	Csm	3.12	3-		-			
1010	BioRe	BioReactor 1 Inf	3.3.13	0644	CSM	8m	<i>†</i>		*		<b>+</b>	
Suur Oss	BioRe	BioReactor 2 Inf	3.8-13	5590	CSW	EM	N		*			
Mengorida	BioRea	BioReactor 2 Eff	3-4-15	0990	esm	3/11	N		7			
de alaid	E	Filter Blk	3-8-13	0320	csm	Bira						
N COM	Meta	Metals Trip Blk	0-18	800	800	range	3		3 444			
C SU							Filtering o	of solubl	e Se pe	Filtering of soluble Se performed in the field	ne field	
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11 Seal/Locked By	Date/Time		12)Sealflock-O	Opened By			)				3-2	
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CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM **Duke Energy Analytical Laboratory Analytical Laboratory Use Only** Duke <sup>19</sup>Page 1 of 2 MATRIX OTHER Samples Mail Code MGO3A2 (Building 7405) Energy 13339 Hagers Ferry Rd DISTRIBUTION Huntersville, N. C. 28078 ORIGINAL to LAB, Logged By Date & Time (704) 875-5245 SAMPLE PROGRAM Ground COPY to CLIENT Fax: (704) 875-4349 **NPDES** Drinking Water 1)Project Name 2)Phone No: Allen - FGD UST\_\_\_ RCRA Waste \_\_\_\_ WWTS (2010, Bi-Monthly Sampling) Cooler Temp (C) 2) Client: 4)Fax No: 15Preserv.:1=HCL Don Scruggs, Robbin Jolly, 2=H2SO4 3=HNO3 Ray Lidke, Bill Kennedy 4 3,4 3,4 4=Ice 5=None 5)Business Unit: 6)Process: Mail Code: MR# 8)Oper. Unit: 9)Res. Type: 10)Resp. Center: Customer to complete all appropriate non-shaded areas. oluble Sampling conducted: 2nd and 4th Monday LAB USE ONLY Se Speciation Bottle Br (D Metal Se, ID <sup>13</sup>Sample Description or ID 11 Lab ID Date Time Signature FGD Purge Eff 3-8-18 0637 CSM BM EQ Tank Eff. 3-8-13 0640 CSM 3-8-13 0644 BioReactor 1 Inf 1 \*\* BioReactor 2 Inf 3-8-13 0655 CSM BM 4\*\* 20 BioReactor 2 Eff 3-8-13 0650 CSM 13/M2 3-8-13 0820 Filter Blk BAR CSM Metals Trip Blk 4 \*\* Filtering of soluble Se performed in the field Customer to sign & date below - fill out from left to right. 1) Relinquished By Date/Time 2) Accepted By <sup>22</sup>Requested Turnaround 3) Relinquished By Date/Time 4) Accepted By Date/Time 14 Days 5)Relinquished By Date/Time 6)Accepted By: Date/Time \*7 Days 7)Relinquished By 8)Accepted By: Date/Time • 48 Hr 9)Seal/Locked By 10) Seal/Lock Opened By Date/Time \*Other \* Add. Cost Will Apply 11)Seal/Locked By Date/Time 12)Seal/Lock Opened By Date/Time Comments

B by TRM/ICP

1\*\*=No Hg analyzed

\* Metals=As, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS,

# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM **Duke Energy Analytical Laboratory Analytical Laboratory Use Only** Duke Energy su <sup>19</sup>Page 2 of 2 MATRIX OTHER Samples Mail Code MGO3A2 (Building 7405) Originating DISTRIBUTION 13339 Hagers Ferry Rd ORIGINAL to LAB, Huntersville, N. C. 28078 3-12-13 Logged By SAMPLE PROGRAM Ground COPY to CLIENT (704) 875-5245 Water \_\_\_\_ NPDES Fax: (704) 875-4349 **Drinking Water** Vendor B&RAND 2)Phone No: 1)Project Name Allen - FGD RCRA Waste \_\_\_\_ WWTS (2011, Bi-Weekly Sampling) Cooler Temp (C) 4)Fax No: 15Preserv.:1=HCL IPO# 2) Client: Bill Kennedy, Robbin Jolly, Don Scruggs, 2=H2SO4 3=HNO3 Ray Lidke 4=Ice 5=None MR# 5)Business Unit: 6)Process: Mail Code: 9)Res. Type: 8)Oper. Unit: 10)Reso. Center: Customer to complete all appropriate non-shaded areas. Sampling conducted: 2nd Monday each month LAB USE ONLY Se Speciation Bottle <sup>13</sup>Sample Description or ID 11 Lab ID Date Time Signature CSM BM BioReactor 1 Inf 3-8-13 0720 Hg Blk BioReactor 1 Inf 3-8-13 0720 BioReactor 2 Inf CSM 3-8-13 0727 Hg Blk BioReactor 2 Inf 38-13 0727 3-8-13 0724 CSM B/R BioReactor 2 Eff Hg Blk BioReactor 2 Eff 3-8-13 0724 CSM Use the Bioreactor 2 Inf or Eff sample as the MS/MSD Customer to sign & date below - fill out from left to right. 1) Relinquished By Date/Time 2) Accepted By Date/Time <sup>22</sup>Requested Turnaround 3) Relinquished By Date/Time 4) Accepted By Date/Time 14 Days 5)Relinquished By Date/Time 6)Accepted By: Date/Time \*7 Days 7)Relinquished By 8)Accepted By: Date/Time - 48 Hr 9)Seal/Locked By 10) Seal/Lock Opened By Date/Time \*Other\_\_\_\_ \* Add. Cost Will Apply 11)Seal/Locked By Date/Time 12)Seal/Lock Opened By Date/Time Comments